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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

WASHINGTON, D.C. 20546

REPLY TO
ATTN OF:

GP

DEC 2 1974

TO: KSI/Scientific & Technical Information Division
Attn: Miss Winnie M. Morgan

FROM: GP/Office of Assistant General
Counsel for Patent Matters

SUBJECT: Announcement of NASA-Owned U.S. Patents in STAR

In accordance with the procedures agreed upon by Code GP and Code KSI, the attached NASA-owned U.S. Patent is being forwarded for abstracting and announcement in NASA STAR.

The following information is provided:

U.S. Patent No. : 3,847,115
Government or : U.S. Government
Corporate Employee
Supplementary Corporate : [Signature]
Source (if applicable)
NASA Patent Case No. : MFS-20,775-1

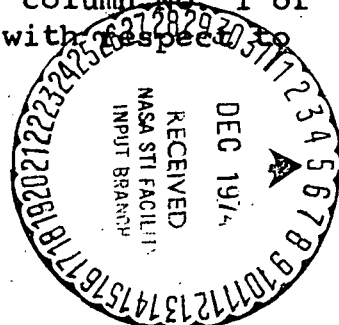
NOTE - If this patent covers an invention made by a corporate employee of a NASA Contractor, the following is applicable:

YES ☐ NO ☒

Pursuant to Section 305(a) of the National Aeronautics and Space Act, the name of the Administrator of NASA appears on the first page of the patent; however, the name of the actual inventor (author) appears at the heading of column No. 1 of the Specification, following the words "...with respect to an invention of ..."

Bonnie L. Woerner

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Enclosure



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(NASA-Case-MFS-20775-1) SYSTEM FOR
DEPOSITING THIN FILMS Patent (NASA)
6 p

United States Patent [19]

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Tashbar

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[54] SYSTEM FOR DEPOSITING THIN FILMS

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[73] Assignee: The United States of America as represented by the Administrator of the National Aeronautics and Space Administration, Washington, D.C.

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[52] U.S. Cl. 118/49.1

[51] Int. Cl. C23c 13/08

[58] Field of Search 118/7, 8, 49.1, 49.5; 117/93.3; 204/298

[56] References Cited

UNITED STATES PATENTS

3,294,583 12/1966 Fedotowsky 117/93.3 X

3,341,352	9/1967	Ehlers	117/93.3
3,573,098	3/1971	Bieber et al.	117/93.3 X
3,654,109	4/1972	Hohl et al.	204/298
3,669,860	6/1972	Knowles et al.	204/298 X
3,679,571	7/1972	Ensslin	204/298

Primary Examiner—Morris Kaplan

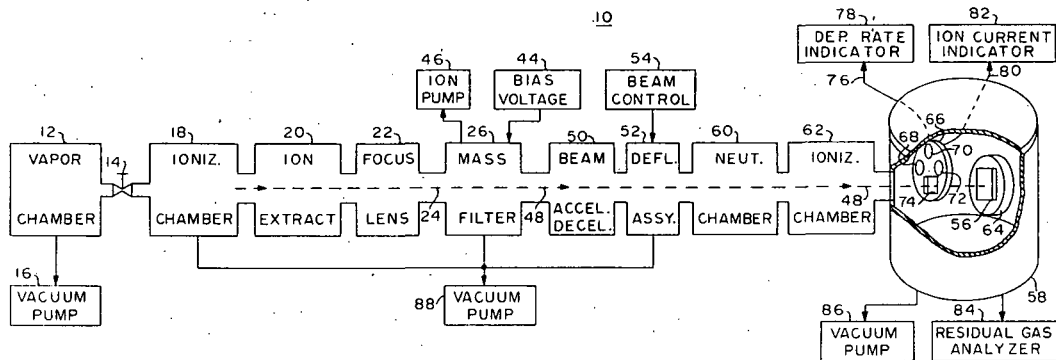
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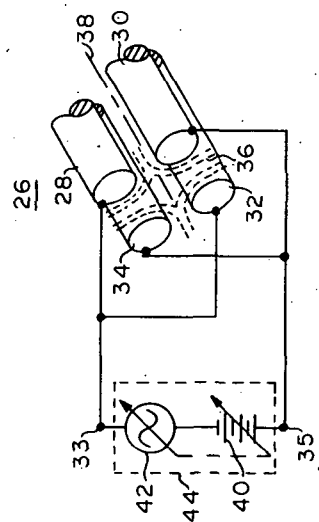
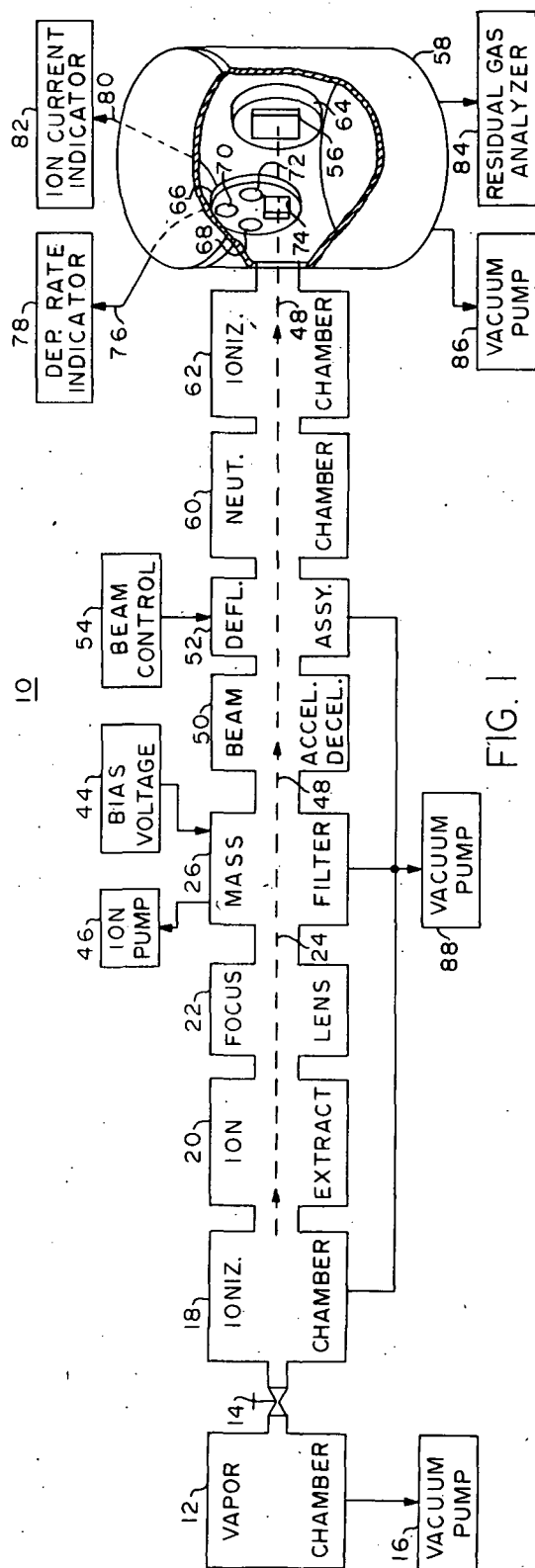
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ABSTRACT

A system for depositing a thin film of one material on another in which a mass filter is interposed between the source of material being deposited and object upon which deposition is being made.

3 Claims, 2 Drawing Figures





SYSTEM FOR DEPOSITING THIN FILMS

ORIGIN OF THE INVENTION

The invention described herein was made by an employee of the United States Government and may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

BACKGROUND OF THE INVENTION

1. Field of the Invention:

This invention relates to systems and devices for depositing thin layers of one material on another.

2. General Description of the Prior Art:

Two methods are most commonly employed in the deposition of thin films. In one, referred to as the cathode sputtering method, a potential difference is applied across two electrodes in a selected gas environment, thereby setting up a cold discharge. A metal to be deposited is used as the cathode electrode and the substrate which is to receive metal atoms from the cathode is positioned between the cathode and anode electrodes of the system. The ions from the cathode are ejected by the incident of positive ions in the resulting gases discharge.

In the other method, an evaporation method, a filament crucible or electron beam is used to heat a material to be deposited. The ejected atoms or molecules are then deposited on a substrate within a vacuum chamber at some distance from the filament crucible or evaporator.

A number of difficulties have arisen in utilizing known methods and it has been found that these difficulties include one or more of the following factors:

1. Objects with complex surface contours are difficult to coat evenly.
2. Atoms or molecules leave the source at all angles, therefore not all of the material available for deposition impinges upon the substrate to be coated.
3. Clustering of the deposit can occur if the heating of an evaporator is unsatisfactory.
4. Deposition rates are difficult to control.
5. The temperature of the substrate may become undesirably overheated, particularly if it is close to a source which under-goes prolonged heating.
6. Residual gases are sometimes released during a heating cycle and contaminate the substrate.
7. High speed pumps must be employed to overcome outgassing and pressure bursts from certain source materials.
8. Where the substrate is one of the plastic materials it will outgas or give off moisture when exposed to radiant heat from an evaporator source.
9. A monolayer cannot be deposited while varying the components of the source.
10. A source material cannot be precisely doped with impurities.
11. It is difficult and almost impossible to keep pressures below the desired 10-6 torr when depositing.
12. Changes in source material usually require that the system be brought back up to atmospheric pressure before making a change.

SUMMARY OF THE INVENTION

Accordingly, it is the object of this invention to pro-

vide an improved system for the deposition of a thin layer of one material upon another and to overcome the difficulties set forth above with respect to previously known methods and systems.

These and other objects are accomplished in the present invention in which a source of deposition material in vapor form is ionized, focused into a stream, passed through a mass filter which extracts undesired impurities or foreign material, the beam accelerated or decelerated and directly deflected onto a desired position on a substrate material positioned in a vacuum chamber.

BRIEF DESCRIPTION OF THE DRAWING

The invention will be more fully understood by the following detailed description when considered together with the accompanying drawing in which:

FIG. 1 is a diagrammatic illustration of a system constructed in accordance with the invention.

FIG. 2 is a pictorial representation of the construction of a mass filter employed in the system shown in FIG. 1.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring initially to FIG. 1, there is shown a deposition system 10 for applying a thin coating or film of one material on another. A conventional vapor chamber 12 is adapted to provide at its outlet a selected material, in vapor form, at a selected vapor pressure and flow rate. Flow rate may be facilitated by valve 14 at the outlet of vapor chamber 12. Vacuum pump 16 connects to vapor chamber 12 and serves to provide a selected vacuum or vapor pressure in vapor chamber 12 and further serves to remove material from the chamber upon the completion of the deposition cycle.

Vaporized material from vapor chamber 12 or a gas chromatograph (Gc) is applied through valve 14, or a membrane separator, or Frit separator or other known type of separator, at a selected low pressure at a selected rate into ionization chamber 18 which ionizes neutral molecules of the material. Ionization chamber 18 is a conventional device in which, for example, ionization is achieved by electron bombardment from electrons emitted from a heated filament. An ionization chamber employing another method such as field emission, chemical ionization, radio frequency radiation or a capillary arc may alternately be employed.

Next, the charged molecules are electrostatically extracted from ionization chamber 18 and propagated at a selected rate by and through ion extractor 20. Ion extractor 20 is a conventional device employing, for example, a biased gridded aperture. The extracted molecules are then directed through a conventional focusing lens 22 which, for example, may be a single lens Einzel focusing lens.

A single lens Einzel focusing lens generally includes three serially arranged, end to end, tubular elements wherein the entrance element and exit element are biased to a common potential with respect to the middle element. Focusing is accomplished by varying the potential of this bias which varies the focal length of the lens. The function of the lens is to focus the ion or molecular beam of material into an essentially coherent molecular or ion stream 24 which is directed into mass filter 26.

Mass filter 26 is preferably of a high resolution type such as the quadrupole mass filter shown in FIG. 2. This filter, which is of the general type employed in Electronic Associates Model 250B Quadrupole Laboratory Mass Spectrometer, consists of four parallel conductive rods 28, 30, 32 and 34 spaced about a common beam path.

Rods 28 and 32 are oppositely positioned and electrically connected together to one terminal 33 of bias source 44 and oppositely positioned rods 30 and 34 are connected to the other terminal 35 of source 44. This creates a hyperbolic field 36 about beam axis 38 as shown in FIG. 2. Bias source 44 basically includes a radio frequency source 42 and direct current voltage source 40 which are connected in series to provide at terminals 33 and 35 a direct current voltage wherein the direct current voltage to radio frequency voltage ratio is approximately 0.16/1. The amplitude of the output voltage of bias voltage source 44 is variable as illustrated and mass filter 26 functions to permit the passage of molecules of a particular mass-to-charge ratio which is determined by the output voltage of bias source 44. Thus by means of mass filter 26, only ions of a particular species of molecules, those having the selected mass-to-charge ratio, are permitted to transit the filter and thus be utilized. This occurs by virtue of the fact that the selected species tends to oscillate with a relatively low amplitude and thus continues as beam 48, whereas particles having other mass-to-charge ratios tend to oscillate at greater amplitudes and are ejected from field 36 and pumped out of the system by ion pump 46.

A sharply defined beam 48 of selected molecules from a mass filter 26 is directed at a prescribed velocity by a conventional accelerator-decelerator 50, typically one having a gridded biased aperture, into electrostatic deflection assembly 52.

Deflection assembly 52 is conventional, being of the type commonly employed in cathode ray tubes, having opposed pairs of deflection electrodes biased in a conventional manner by bias or beam control 54 to selectively deflect ion beam 48 in a desired geometric pattern on to substrate 56 within vacuum chamber 58.

After being desirably deflected by deflection assembly 52, ion beam 48 may then be completely neutralized, if desired, by neutralizing chamber 60. Neutralizing chamber 60 is typically charged with low velocity electrons from a heated filament, with electrons are allowed to drift into transiting molecules and thus to neutralize any residual positive charge thereon. Neutralization may be particularly required when the surface upon which the deposition is to be made is an insulator such as in the case of plastic, quartz, etc., or when an insulating coating is to be applied upon a conductive material. Under these conditions a positive space charge tends to repel or disperse a positively charged beam.

Conversely the beam may be ionized to a measured degree within a second ionization chamber 62. Ionization chamber 62 preferably utilizes a source of ultraviolet or X-ray radiation to excite beam 48. In this manner a measured amount of radiation can be introduced into beam 48 so as to bring it to a predetermined level of excitation.

Finally, the suitably conditioned beam 48 is directed into vacuum chamber 58.

Vacuum chamber 58 includes substrate mount 64 which is adjustably attached to the inner wall of chamber 58, by means not shown, and is centrally positioned in line of sight of a shaped outlet opening of mass filter 26. Substrate mount 64 is typically heated or cooled in accordance with the requirements of a particular deposition operation upon substrate 56.

A rotatable disc 66, disposed between the outlet of ionization chamber 62 and substrate mount 64 provides selective gating and calibration of ion beam 48. Disc 66 includes radially spaced, conventional, quartz-crystal microbalance 68, Faraday cup 70, electron multiplier 72, schematically shown, and an opening 74 through which the filtered and calibrated beam 48 is directed upon the surface of substrate 56.

A conventional Quartz Crystal Microbalance 68 is utilized to measure deposition rate, its output being connected through lead wires 76 to external deposition rate indicator 78 such as an appropriately calibrated electrical voltmeter.

An enclosed Electron Multiplier 72 and enclosed Faraday cup 70 are used to measure ion current and have their outputs selectively connected through lead wires 80 to external ion current indicator 82, such as an appropriately calibrated voltmeter.

The interior wall of vacuum chamber 58 is typically lined with a liquid nitrogen shroud, not shown, or is cooled by other suitable refrigeration in order to partially trap unwanted gases of ionized spectra.

The selected ion beam 48 or other mass entering vacuum chamber 58 during a deposition operation is constantly monitored by residual gas analyzer 84, such as a conventional mass spectrometer, which also provides an indication of any contaminants within the vacuum system due, for example, to insufficient cleaning. In order to facilitate its cleaning, vacuum chamber 58 is provided with a separate vacuum pump 86 and it may be isolated from ionization chamber 62 by a valve, not shown. The stages of system 10 from the first ionization chamber 18 through the second ionization chamber 62 inclusive, are preferably enclosed within interconnected tubular sections. The walls of each section are refrigerated and thus act as a cryopump which cooperates with ion pump 46 to assist in the removal of unwanted ionized gases or particles which are selectively rejected by mass filter 26.

Although these stages are thus enclosed within a common vacuum, in operation there is developed some difference in pressure between them. Accordingly, inlet connections are provided to vacuum pump 88, which are coupled to first ionization chamber 18, mass filter 26, and deflection assembly 52. In general a selected decreasing pressure gradient is provided from the first to the last stage of the system.

However, since the system will operate at a pressure of 10^{-5} torr, pumpout is really not a problem. If desired, the various stages may be separated by a valve so that each may be separately baked and evacuated.

OPERATION

In operation, selected amounts of vaporized materials from vapor chamber 12 are introduced into first ionization chamber 18. Herein the neutral molecules of a constituent are ionized. The ionized particles are electrostatically extracted from ionization chamber 18 by extractor 20 and directed through electrostatic focusing lens system 22 which forms the particles into a

coherent ion beam 24 and directs them through mass filter 26.

Mass filter 26 is selectively controlled by variable potential source 44 to pass a beam 48 composed of only the desired species of molecules of the material to be deposited. All other particles, that is particles having a different mass to charge ratio, are trapped by mass filter 26. The filtered ion beam 48 is then directed at a selected velocity by beam accelerator-decelerator 50 into vacuum chamber 58.

Before the molecular mass or beam 48 is allowed to deposit upon substrate 56 the deposition rate, ion current, and velocity are calibrated as follows:

Rotary disc 66 is first positioned as that beam 48 is directed either into electron multiplier 72 or Faraday cup 70, as determined by the beam intensity and velocity and then the ion current is adjusted by an appropriate bias control to a proper value. Next, rotary disc 66 is positioned so that the beam strikes quartz crystal microbalance 68 and the predetermined deposition rate is calibrated by monitoring the frequency shift of the Quartz crystal of the microbalance and by adjustment of the amplitude of peak-to-peak output voltage. Then, assuming that beam 48 is suitably ionized, no electrons are released within neutralizing source 60, and no X-ray or ultra-violent energy is injected into second ionization chamber 62.

The desired deposition pattern is effected by applying appropriate deflection voltages from beam control 54 to deflection assembly 52, after which rotary disc 66 is positioned to allow the beam 48 to pass through opening 74 of disc 66 and thus impinge upon substrate 56. Residual gas analyzer 84 monitors the background gases of vacuum chamber 58 to insure that the correct material is being deposited and that chamber 58 contains no undesired elements. In order to terminate the deposition cycle, rotary disc 66 is returned to a monitoring position which interrupts the flow of material to substrate 56, and also positions disc 66 to calibrate the ion beam 48 for a subsequent deposition operation.

What is claimed is:

1. A system for depositing a thin layer of material on an object comprising:

source means including a vapor chamber having an outlet and means for providing a selected material in vapor form at said outlet;

ionization means for receiving said material in vapor form from said outlet of said vapor chamber and ionizing said material;

mass filter means having a flow path therethrough for passing ionized molecules having a selected mass-to-charge ratio unimpeded along said flow path and removing other ionized molecules from said flow

path;

two pairs of spaced, parallel rods wherein a plane symmetrically intercepting the longitudinal axis of said rods of a first pair of said rods intersects at a line a plane symmetrically interconnecting the rods of the other said pair of rods and said line generally defines said flow path of said mass filter means;

flow control means comprising a source of selectable amplitude voltage of combined direct and radio frequency current, one polarity of said voltage being connected to said first pair of rods and the other polarity of said voltage being connected to said second pair of rods;

whereby a selected amplitude of said voltage is applied to said rods enabling ionized molecules having a selected mass-to-charge ratio to proceed along said flow path and to completely transit said mass filter means and enabling the removal of other molecules from said flow path;

flow control means for receiving ionized molecules from said ionization means and directing them along said flow path of said mass filter;

a deposition chamber adapted to contain an object upon which a selected material is to be deposited; and

flow directional means responsive to the output flow of ionized molecules passed by said mass filter for directing ionized molecules along selected paths into said deposition chamber and onto selected areas of said object;

whereby precisely selected molecules of a material may be deposited on precisely selected areas of a said object.

2. A system as set forth in claim 1 further comprising deposition rate indicating means including means selectively positionable within the flow path of said ionized molecules and including means responsive to the interception of said flow of material for indicating the magnitude of flow, and thereby indicating the rate of deposition to be anticipated by said flow.

3. A system as set forth in claim 2 comprising:

first vacuum means for applying a selected degree of vacuum to said vapor chamber;

second vacuum means for applying a selected degree of vacuum to said flow control means, said mass filter means and said flow directional means; and

third vacuum means for applying a selected degree of vacuum to said deposition chamber;

whereby a selected pressure gradient is provided along the flow path of molecules transiting said system.

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